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Mr. Nestor J. Camara
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Re: West, TX

B3SA2525
Considerations for Testing Protocol

Armstrong Forensic Laboratory, Inc. (Armstrong) is providing a list of tests including a brief summary of each method for your consideration. Armstrong is not specifically recommending any of the tests, as each of the involved parties may have different objectives. Many of the procedures and tests will be conducted at the Armstrong facility and are identified as such. Several of the tests listed require specialized equipment and will be performed by an outside laboratory. Suitable outsourcing laboratories will be located if these tests are requested.

The laboratory testing for this project has been divided into four (4) categories:

Test Series A: Material Specifications

Test Series B: Reactive/Thermal Characteristics

Test Series C: Composition/Contaminants

Test Series D: Particle Characteristics

Test Series A: Material Specifications:

A1: Bulk Density: USP <616>, Method I—Measurement In A Graduated Cylinder; Yazoo City Nitrogen Complex Procedure No. 113-WI-014

Approximately 100 grams of test sample is introduced into a dry graduated cylinder, without compacting, (M) and the unsettled apparent volume is read (V_0). The bulk density is calculated in g/ml by the formula M/V_0.

Estimated minimum sample quantity required: 200 grams

Analysis is non-destructive

Test is performed by Armstrong

A2: % Fines: ASTM D422
ASTM D422 - 63(2007) Standard Test Method For Particle-Size Analysis Of Soils

This test method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 micrometers (retained on the no. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 micrometers is determined by a sedimentation process using a hydrometer. The balances, stirring apparatus, hydrometer, sedimentation cylinder, thermometer, sieves, water bath or constant-temperature room, beaker, and timing device used in the method are specified. Sieve analysis, hydrometer analysis, and hygroscopic moisture analysis would be performed on the sample soil.

Estimated minimum sample Quantity Required: 100 Grams
Analysis is considered destructive
Test is performed by an outside laboratory

A3: Angle Of Repose: Yazoo City Nitrogen Complex Procedure No. 113-WI-015

The angle of repose of a bulk solid is determined by forming a conical pile of the sample on a flat surface, photographing the pile and measuring the angle of the cone from the photograph

Estimated minimum sample quantity required: 1000 grams
Analysis is non-destructive
Test is performed by Armstrong

A4: Total Acidity: EPA 305.1

The pH of the sample is determined. The sample is titrated electrometrically with standard alkali (sodium hydroxide) to pH 8.2.

Note: The method will be modified for solids
Estimated minimum sample quantity required: 25 grams
Analysis is destructive
Test is performed by Armstrong

A4.1 Corrosivity/pH : EPA 9045D - Soil and Waste pH

This method is an electrometric procedure for measuring pH in soils and waste samples.

Estimated minimum sample quantity required: 25 grams
Analysis is destructive
Test is performed by Armstrong

A5: % Nitrate: EPA 300.0, Ion Chromatography; Yazoo City Nitrogen Complex Procedure No. 113-WI-018

For EPA 300.0, a solution of the solid is prepared and a small volume is introduced into an ion chromatograph. The anions of interest are separated and the concentration of nitrate is measured. The sample will be prepared as described in Procedure No. 113-WI-018.

Estimated minimum sample quantity required: Less Than 5 Grams
Analysis is destructive
Test is performed by Armstrong

A6: (Ammonia Nitrogen) NH3-N: ASTM D6919, Ion Chromatography; Yazoo City Nitrogen Complex Procedure No. 113-WI-019

For ASTM D6919, a solution of the solid is prepared and a small volume is introduced into an ion chromatograph. The cations of interest are separated and the concentration of ammonium is measured. The sample will be prepared as described in Procedure No. 113-WI-019.

Estimated minimum sample quantity required: Less Than 5 grams
Analysis is destructive
Test is performed by Armstrong

A7: % Nitrogen: Calculation From A5 And A6

A8: % Ammonium Nitrate: Calculation From A5 And A6

A9: Friability: Yazoo City Nitrogen Complex Procedure 113-WI-016

The only known facility where this analysis is performed is the Yazoo City Nitrogen Complex. This method is application-specific, and interpretation may not be straightforward.

Estimated minimum sample quantity required: 250 grams
Analysis is destructive
Test would be performed at Yazoo City Nitrogen Complex

Test Series B – Reaction/Thermal Characteristics

B1: Corrosivity/pH: EPA 9045D - Soil and Waste pH

This method is an electrometric procedure for measuring pH in soils and waste samples.

Estimated minimum sample quantity required: 25 grams
Analysis is destructive
Test is performed by Armstrong

B2: Corrosivity Toward Steel: EPA 1110A

The test exposes coupons of SAE Type 1020 steel to an aqueous solution of the material and by measuring the degree to which the coupon has been dissolved, determines the corrosivity of the material.

Estimated minimum sample quantity required: 100 grams

Analysis is destructive

Test is performed by Armstrong

B3: Thermal Stability: ASTM E487-09, Constant Temperature Stability Of Chemical Materials; ASTM E537-07, Thermal Stability Of Chemicals By Differential Scanning Calorimetry

B3.1 Standard Test Method For Constant-Temperature Stability Of Chemical Materials

This test method describes the assessment of constant temperature stability of chemical materials that undergo exothermic reactions. The techniques and apparatus described may be used on solids, liquids, or slurries of chemical substances.

When a series of materials is tested by this test method, the results permit ordering the materials relative to each other with respect to their thermal stability.

Limitations of test:

This test method is limited to ambient temperatures and above.

This test method determines neither a safe storage temperature nor a safe processing temperature.

Note: A safe storage or processing temperature requires that any heat produced by a reaction be removed as fast as it is generated and that proper consideration be given to hazards associated with reaction products.

Estimated minimum sample quantity required: 15 grams

Analysis is destructive

Test is performed by an outside laboratory

B3.2 Thermal Stability DSC/TGA: ASTM E537-07 Standard Test Method For The Thermal Stability of Chemicals by Differential Scanning Calorimetry

This test method describes the ascertainment of the presence of enthalpic changes in a test specimen, using minimum quantities of material, approximates the temperature at which these enthalpic changes occur and determines their enthalpies (heats) using differential scanning calorimetry or pressure differential scanning calorimetry.

This test method may be performed on solids, liquids, or slurries. This test method may be performed in an inert or a reactive atmosphere with an absolute pressure range from 100 Pa through 7 MPa and over a temperature range from 300 to 800 K (27 to 527°C).

Estimated minimum sample quantity required: 15 grams

Analysis is destructive

Test is performed by an outside laboratory

B4: Spontaneous Combustion: EPA 1050 - Test Methods to Determine Substance Likely to Spontaneously Combust

This method provides test procedures, which may be used to evaluate and categorize liquid and solid wastes that are likely to spontaneously combust. The method is based on the DOT regulations for the transport of spontaneously combustible materials as provided in 49 CFR part 173, appendix e. These test procedures are intended to identify two types of wastes with spontaneous combustion properties: wastes (including mixtures and solutions, liquid or solid) which, even in small quantities, ignite within five minutes of coming in contact with air. These wastes are the most likely to spontaneously combust and are considered to have pyrophoric properties. Other solid wastes which, in contact with air and without an energy supply, are susceptible to self-heating. These wastes will ignite only when in large amounts (kilograms) and after long periods of time (hours or days) and are considered to have self-heating properties.

Test Method C -- Self-Heating Wastes

Test method C may be performed to determine if a solid waste exhibits self-heating properties. This test procedure is limited to granular solids, pastes, and other solid wastes that can be reduced in particle size to fit into a 25-mm or 100-mm stainless steel cube.

In a preliminary test, a 100-mm sample cube [filled with the granular material] is exposed to a test temperature of 140 ± 2°C for 24 hours and the sample is observed to determine if it undergoes spontaneous ignition or a rise in sample temperature to over 200°C within 24 hours. If the results of the preliminary test are positive, a second test using a 25-mm sample cube is conducted to further classify the waste.

The presence of any smoke, incandescence, or flame under the test conditions provided should be interpreted as a positive result for spontaneous heating. A positive result for test method C is demonstrated by a significant increase in sample temperature. The analyst should be consistent with ignition recognition and record any visible signs of combustion including smoke, heat, flame, or incandescence. Ignition in any of the trials is considered a positive result, even when the results of the other trials are negative.

Estimated minimum sample quantity required: 2 kilograms

Analysis is destructive

Test is performed by Armstrong

B5: Minimum Autoignition Temperature (MAIT) of Dust Clouds: ASTM E1491-06.

This test method covers the determination of the minimum temperature at which a dust cloud will autoignite. The dust cloud is exposed to air heated to various temperatures in a furnace. Ignition of the cloud is indicated by visual observation of the flame. The chemical nature of the dust as well as its concentration, particle size, moisture, and surface area affects the results.

Estimated Minimum Sample Quantity Required: 2 kilograms

Analysis is destructive

Test is performed by an outside laboratory

B6: Drop Weight Sensitivity Of Solid Phase Hazardous Materials: ASTM E680-79

This test method is designed to determine the relative sensitivities of solid-phase hazardous materials to drop weight impact stimulus.

Restrictions are placed upon the ranges of impact tool masses and striking surface diameters that may be used, and a standard sample thickness is prescribed for all tests. In addition, procedures for sample

preparation and treatment, as well as procedures for detecting reactions through the use of the human senses, are outlined.

Drop-weight impact tests are to be performed using the well-known bruceton up-and-down method.

This test method does not require an overall rigid standardization of the apparatus. Samples are tested either unconfined or confined in confinement cups. For confined tests, some of the important cup parameters, such as cup material, cup wall thickness, and fit between the cup and the striking pin, are standardized. Data generated from unconfined and confined tests will not, in general, exhibit the same relative scale of sensitivities, and must be identified as confined or unconfined data and compared separately.

Estimated Minimum Sample Quantity Required: Unknown

Analysis is destructive

Test is performed by an outside laboratory

B7: Calculation Of Hazard Potential Figures Of Merit For Thermally Unstable Materials: ASTM E1231-10

This practice provides nine figures-of-merit which may be used to estimate the relative thermal hazard potential of thermally unstable materials. Since numerous assumptions must be made in order to obtain these figures-of-merit, care must be exercised to avoid too rigorous interpretation (or even misapplication) of the results.

This practice may be used for comparative purposes, specification acceptance, and research. It should not be used to predict actual performance.

This practice covers the calculation of hazard potential figures-of-merit for exothermic reactions, including:

(1) Time-to-thermal-runaway,

(2) Time-to-maximum-rate,

(3) Critical half thickness,

(4) Critical temperature,

(5) Adiabatic decomposition temperature rise,

(6) Explosion potential,

(7) Shock sensitivity,

(8) Instantaneous power density, and

(9) NFPA instability rating.

B8: Porosity/Surface Morphology: No Methodology Selected

The surface morphology of the material may be examined via Scanning Electron Microscopy (SEM).

Estimated Minimum Sample Quantity Required: 5 grams

Analysis is destructive

Test is performed by an outside laboratory

Test Series C – Composition/Contaminants

C1: Total Metals: EPA 6010C, Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-OES)

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) may be used to determine trace elements in solution. Solid matrices need acid digestion prior to analysis. The following analytes have been determined by this method:

Element	Symbol	CAS Number	Element	Symbol	CAS Number
Aluminum	Al	7429-90-5	Manganese	Mn	7439-96-5
Antimony	Sb	7440-36-0	Molybdenum	Mo	7439-98-7
Arsenic	As	7440-38-2	Nickel	Ni	7440-02-0
Barium	Ba	7440-39-3	Phosphorus	P	7723-14-0
Beryllium	Be	7440-41-7	Potassium	K	7440-09-7
Boron	B	7440-42-8	Selenium	Se	7782-49-2
Cadmium	Cd	7440-43-9	Silver	Ag	7440-22-4
Calcium	Ca	7440-70-2	Sodium	Na	7440-23-5
Chromium	Cr	7440-47-3	Strontium	Sr	7440-24-6
Cobalt	Co	7440-48-4	Thallium	Tl	7440-28-0
Copper	Cu	7440-50-8	Tin	Sn	7440-31-5
Iron	Fe	7439-89-6	Titanium	Ti	7440-32-6
Lead	Pb	7439-92-1	Vanadium	V	7440-62-2
Lithium	Li	7439-93-2	Zinc	Zn	7440-66-6
Magnesium	Mg	7439-95-4			

(CAS Number: Chemical Abstract Service Registry Number)

Estimated Minimum Sample Quantity Required: 10 grams

Analysis is destructive

Test is performed by Armstrong

C2: Mercury: EPA 7471B, Cold Vapor Atomic Absorption

This method is a cold-vapor atomic absorption procedure for measuring total mercury. All samples must be subjected to an appropriate dissolution step prior to analysis. If this dissolution procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix.

Estimated Minimum Sample Quantity Required: 10 grams

Analysis is destructive

Test is performed by Armstrong

C3: X-Ray Diffraction: Analysis Of Solids For Molecular Identification (Crystalline Material Only)

Estimated Minimum Sample Quantity Required: 10 grams

Analysis is destructive

Test is performed by Armstrong

C4: SEM/EDX: Analysis Of Solids For Elemental Identification

Estimated Minimum Sample Quantity Required: 5 grams

Analysis is destructive

Test is performed by an outside laboratory

C5: Moisture Analysis: ASTM D2216

These test methods cover the laboratory determination of the water (moisture) content by mass of soil, rock, and similar materials where the reduction in mass by drying is due to loss of water

Estimated Minimum Sample Quantity Required: 10 grams

Analysis is destructive

Test is performed by Armstrong

C6: Total Hexane Extractables/Oil & Grease/Fuel Oil: EPA 9071B

Method 9071 may be used to quantify low concentrations of oil and grease in soil, sediments, sludges, and other solid materials amenable to chemical drying and solvent extraction with n-hexane. "Oil and grease" is a conventional pollutant under 40 CFR 401.16 and generally refers to substances, including biological lipids and mineral hydrocarbons, that have similar physical characteristics and common solubility in an organic extracting solvent. As such, oil and grease is an operationally defined parameter, and the results will depend entirely on the extracting solvent and method of extraction. Method 9071 employs n-hexane as the extraction solvent with soxhlet extraction and the results of this method are appropriately termed "n-hexane extractable material (HEM)."

Estimated Minimum Sample Quantity Required: 100 grams

Analysis is destructive

Test is performed by Armstrong

C7: Volatile Organic Compounds: EPA 8260, Gas Chromatography/Mass Spectrometry (GC/MS) Analysis

Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices.

Estimated Minimum Sample Quantity Required: 10 Grams

Analysis is destructive

Test is performed by Armstrong

C8: Semi-Volatile Organic Compounds: EPA 8270, Gas Chromatography/Mass Spectrometry (GC/MS) Analysis

This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples.

Estimated Minimum Sample Quantity Required: 10 Grams

Analysis is destructive

Test is performed by Armstrong

C6: Bulk Density: USP <616>, Method I—Measurement In A Graduated Cylinder; Yazoo City Nitrogen Complex Procedure No. 113-WI-014

Approximately 100 grams of test sample is introduced into a dry graduated cylinder, without compacting, (M) and the unsettled apparent volume is read (V_0). The bulk density is calculated in g/ml by the formula M/V_0.

Estimated minimum sample quantity required: 200 grams

Analysis is non-destructive

Test is performed by Armstrong

C7: Anions: EPA 300.0, Ion Chromatography

For EPA 300.0, a solution of the solid is prepared and a small volume is introduced into an ion chromatograph. The anions of interest are separated and measured. The sample will be prepared as described in Yazoo City Nitrogen Complex Procedure No. 113-WI-018.

Target Anions: Bromide, Chloride, Nitrate, Nitrite, Phosphate, and Sulfate

Estimated minimum sample quantity required: Less Than 5 Grams

Analysis is destructive

Test is performed by Armstrong

C8: Cations: ASTM D6919-03, Ion Chromatography

For ASTM D6919, a solution of the solid is prepared and a small volume is introduced into an ion chromatograph. The cations of interest are separated and measured. The sample will be prepared as described in Yazoo City Nitrogen Complex Procedure No. 113-WI-019.

Target Cations: Lithium, Sodium, Ammonium, Potassium, Magnesium, and Calcium

Estimated minimum sample quantity required: Less Than 5 grams

Analysis is destructive

Test is performed by Armstrong

Test Series D – Particle Characteristics

D1: Particle Size Distribution: PM-10/NIOSH P&CAM 239

The examination is performed via optical microscopy. A representative portion of the material is examined and the particle sizes and distributions are measured.

Estimated minimum sample quantity required: 20 grams

Analysis is considered destructive

Test is performed by Armstrong

D2: Particle Size By Sieves: ASTM D422 - Standard Test Method For Particle-Size Analysis Of Soils

This test method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 micrometers (retained on the no. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 micrometers is determined by a sedimentation process using a hydrometer. The balances, stirring apparatus, hydrometer, sedimentation cylinder, thermometer, sieves, water bath or constant-temperature room, beaker, and timing device used in the method are specified. Sieve analysis, hydrometer analysis, and hygroscopic moisture analysis would be performed on the sample soil.

Estimated minimum sample Quantity Required: 100 Grams

Analysis is considered destructive

Test is performed by an outside laboratory

Costs of analytical testing vary greatly. It is based on the complexity of the method, the equipment required and how many samples are being analyzed. A cost estimate can best be determined after the testing is requested.

Respectfully submitted,
Armstrong Forensic Laboratory, Inc.

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